



ARL-MR-0965 • SEP 2017



Optimization of Easy Atomic Force Microscope (ezAFM) Controls for Semiconductor Nanostructure Profiling

by Satwik Bisoi and Naresh Das

Approved for public release; distribution is unlimited.

NOTICES

Disclaimers

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

Citation of manufacturer's or trade names does not constitute an official endorsement or approval of the use thereof.

Destroy this report when it is no longer needed. Do not return it to the originator.



Optimization of Easy Atomic Force Microscope (ezAFM) Controls for Semiconductor Nanostructure Profiling

by Satwik Bisoi

Science and Engineering Apprentice Program, Adelphi, MD

Naresh Das

Sensors and Electron Devices Directorate, ARL

REPORT DOCUMENTATION PAGE				Form Approved OMB No. 0704-0188	
<p>Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing the burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.</p> <p>PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.</p>					
1. REPORT DATE (DD-MM-YYYY) September 2017		2. REPORT TYPE Memorandum Report		3. DATES COVERED (From - To) 2017 July 05–2017 August 18	
4. TITLE AND SUBTITLE Optimization of Easy Atomic Force Microscope (ezAFM) Controls for Semiconductor Nanostructure Profiling				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) Satwik Bisoi and Naresh Das				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) US Army Research Laboratory Sensors and Electron Devices Directorate (ATTN: RDRL-SEE-I) 2800 Powder Mill Road Adelphi, MD 20783-1138				8. PERFORMING ORGANIZATION REPORT NUMBER ARL-MR-0965	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT <p>The US Army Research Laboratory recently procured an Easy Atomic Force Microscope (ezAFM), from a NanoMagnetics vendor. The ezAFM can profile nanostructures on the order of 2.0 Å in the vertical (z) directions. There are several instruments controls like proportional, integral, and derivative (PID) gain as well as tip force and laser power that have to be set right to get best surface profiling of semiconductor nanostructured materials. In this report, we present how to obtain good-quality profiling pictures using ezAFM by varying PID values. We used the equipment supplier-provided standard calibration samples as well as our in-house-grown nanostructure samples.</p>					
15. SUBJECT TERMS ezAFM, Easy Atomic Force Microscope, atomic force microscope, nanostructures, profiling pictures, calibration					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UU	18. NUMBER OF PAGES 16	19a. NAME OF RESPONSIBLE PERSON Naresh Das
a. REPORT Unclassified	b. ABSTRACT Unclassified	c. THIS PAGE Unclassified			19b. TELEPHONE NUMBER (Include area code) (301) 394-5500

Contents

List of Figures	iv
List of Tables	iv
1. Introduction	1
2. Experiment	1
3. Results and Discussion	3
4. Conclusion	5
5. References	7
List of Symbols, Abbreviations, and Acronyms	8
Distribution List	9

List of Figures

Fig. 1	A 2-D scan of the standard grating sample with ezAFM.....	4
Fig. 2	A 3-D image of the grating sample.....	4
Fig. 3	Peak-to-peak analysis of the grating sample in Figs. 1 and 2.....	4
Fig. 4	a) 2-D surface profile of Au nanofilm on silicon substrate, b) 3-D view of same sample, c) average roughness values, and d) roughness of particles in a line	5

List of Tables

Table 1	Descriptions of each step in the ezAFM scanning process.....	3
---------	--	---

1. Introduction

The Easy Atomic Force Microscope (ezAFM) by NanoMagnetics Instruments (Celik et al. 2012) has been used as an alternative to the previous atomic force microscopes (AFMs). The ezAFM provides a more compact apparatus as well as a simpler scanning process, while still providing scans of the topography of materials, just as previous AFMs do. This allows for scans of possible defects of sample surfaces, as well as displays of changes in topography.

In using the ezAFM for topographical scanning, there are 4 main parameters that must be maximized for a high-quality scan. These parameters can be referred to as the PID control, where P stands for proportional gain, I stands for integral gain, and D stands for derivative gain. An additional parameter that contributes to the scanned image quality is set point. Proportional gain is multiplied by the error to adjust controller output and integral gain sums the error history over time, but is the best when at a constant value of 1. Derivative gain counteracts P and I to reduce overshooting and ringing. Set point is defined as a measure of the force applied from the tip to the sample. This value is best when set between 50% and 60%. For each material or sample that is scanned with the ezAFM, the PID control must be varied due to varying surface structures. Finding these ideal values for a high-quality scan requires repeated testing of various values until a basic trend is determined.

2. Experiment

Prior to starting work with the ezAFM, one must accomplish 3 steps: 1) calibrate the vibration isolation platform to minimize the amount of outside noise, 2) make sure the cantilever ready sign is green (otherwise, there is a problem with the AFM tip) and 3) tune the laser to the scanning frequency (using auto-tuning, one should see a sharp peak at a predefined frequency). Various samples were stuck to plates with an adhesive that came with the ezAFM kit, or one can use double-sided tape to mount the samples. It is helpful to smooth out the tape surface such that the sample will be flat when the profile is taken.

To measure if the noise levels in the lab are acceptable, a sample of mica was used for calibration. Mica has an extremely flat surface and is used with the ezAFM to determine relative noise. The mica sample is placed in the ezAFM apparatus and, once set, work proceeds using the ezAFM program on the computing device.

Prior to checking the noise level using the program, one must first open the camera. The camera provides a view of the surface, so one can adjust the cantilever so as

not to damage the surface. Using the x-y motor option, we shifted the cantilever until it was directly above the sample surface.

Next, one must set the program to auto-tune the cantilever; this setting will remain every time the program opens. First, the laser power (FT) must stay so it is at a constant 2 V at all times. Once the FT steadies to a constant 2 V, the excitation slider should be varied to reach an oscillation amplitude (OA) of approximately 2.0 V. We consider it beneficial to start with low OA values and then increase the OA values to optimize PID values to get the best profile picture. Following that, one should select the “Auto Land” option, which allows the cantilever to automatically lower until it reaches the surface. The z-position, which determines the height of the cantilever, was monitored to determine when it started to reach a constant value. When this value was reached (the blue arrow should be at the middle of the approach column), one should press the downwards arrow twice to stop the cantilever’s approach. From here, we set the P and D to the maximum 100, I to the constant value 1, and set point to 50%.

After inputting the scan options (like scan area, scan rate, image size, etc.), the calibration scan was conducted. Immediately following the end of the scan, the cross-sectional tool in the image tools was used. This tool was used by marking a perpendicular line across the entire scan area. This creates a graph on which a range of the values is determined. If the range is less than 2 Å, the noise level is acceptable.

One of the first samples tested was an in-house fabricated gold (Au) nanofilm (5 nm) on a silicon substrate. The same scanning process is applicable for this material, as well as all others tested. At this point, relative patterns or deformities were visible on the surface in the camera screen. The x-y motor can be used to view different areas on the sample’s surface. The scan option and parameters for image size and scan speed were selected. From there, the scan proceeded. During the scan, some stray lines or other imperfections may appear. Using the image tools can help minimize these imperfections. Plane correction can correct the scan to distinguish between the different planes. By looking at the graph for amplitude during the scan, the relative noise can be determined. If high variations in the noise levels are observed, then the P and D values should be decreased during the scan. If the opposite is true, the P and D should be increased.

Once the scan was completed, the relative quality of the image was determined through pure observation. However, it was necessary to gain more information about the quality of the image. The roughness analysis provides an acceptable analysis of the quality of the image by providing the relative roughness of the scan. In addition, other image tools and settings were available to further process each

scan. For instance, a peak-to-peak analysis can be done using the roughness measurement tool in the image analysis section.

Note: If the ezAFM is already calibrated, one can use a Quick Start procedure (NanoMagnetics Instruments 2014): turn on the ezAFM software and the switch in the back of the controller and wait 30 min before using the machine.

The entire ezAFM process is outlined in Table 1.

Table 1 Descriptions of each step in the ezAFM scanning process

Step Name	Procedure	Remarks
Laser on	Adjust the laser power to 70%–80% until the cantilever ready sign is green.	If the cantilever sign is not green, it may be time to replace the AFM tip.
OA	Adjust the setting to get an OA of 2.0 V.	Start with smaller OA numbers.
Tuning	After setting FT power and OA to 2.0 V, click the auto-tune command.	One should see sharp peak and the Q value increases; aim for $Q > 300$.
PID setting	Always start with smaller P and D values, maybe 10%, and keep I at 1.	One can change the P and D values while scanning to minimize noise.
Set point	Keep the set point between 50%–60%.	The set point depends on hardness of the sample.
Camera	Turn on the camera to see both the tips and the surface of the sample.	Use intensity button to control the light for the camera.
Stage	Use the stage x-y move button to select the scan area.	This sets the speed at which stage moves.
Approach	Use the auto landing button to bring the AFM tip close to the sample surface.	Hit the button arrow twice to bring the blue pointing arrow to middle.
Scan	Set the scan parameter: area, rate, and picture quality.	Select one of the pictures and apply an image analysis tool.
Image analysis	Very useful tools to correct and enhance an image.	Right click on the image to save in different formats.

3. Results and Discussion

After testing various samples with the ezAFM, the highest quality images were created using the correct variations of the PID control and image tools. Figure 1 shows the standard grating sample that was tested using the experimental process detailed in Section 2. Figure 2 displays the image analysis data of the same scan.

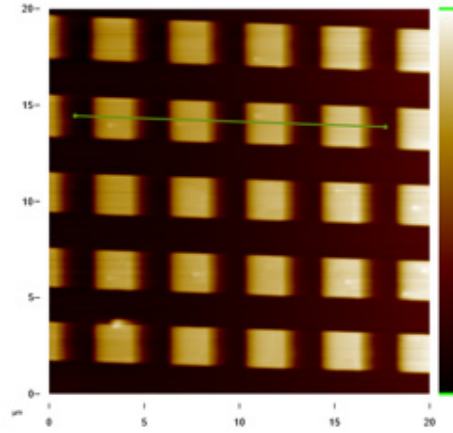


Fig. 1 A 2-D scan of the standard grating sample with ezAFM

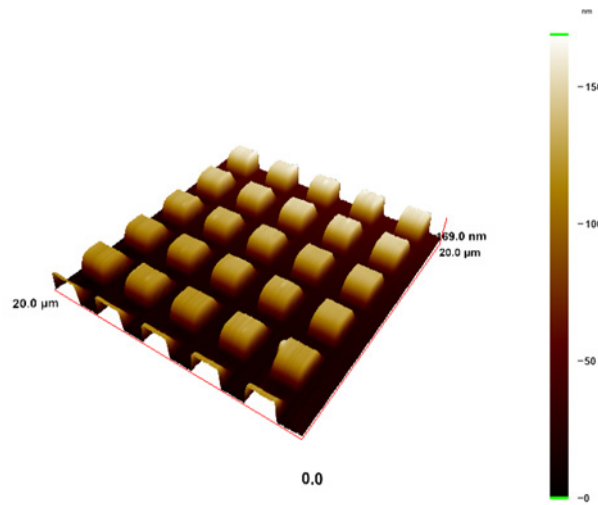


Fig. 2 A 3-D image of the grating sample

Figure 3 displays the peak-to-peak analysis of the grating sample. The grating pattern has metal thickness of 110 nm on average.

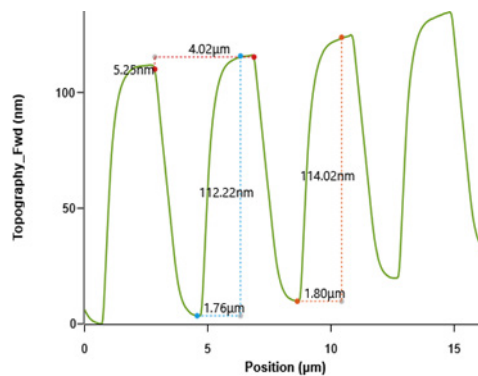


Fig. 3 Peak-to-peak analysis of the grating sample in Figs. 1 and 2

In Fig. 4, we show the AFM scan of an Au nanofilm on a silicon substrate. First, a 5-nm Au film was deposited on a silicon substrate using an e-beam evaporation technique. It was then annealed in an oxygen gas (O_2) environment at 450 °C for 4 h and then the AFM scan was taken. Due to the presence of dust particles, as seen in the bottom-right portion of the 3-D plot in Fig. 4b, we observed a particle height of 17 nm. As seen in Fig. 4d, some of the particles have a height of about 2.5 to 3 nm. The roughness of the film was about 1 nm, as seen in Fig. 4c.

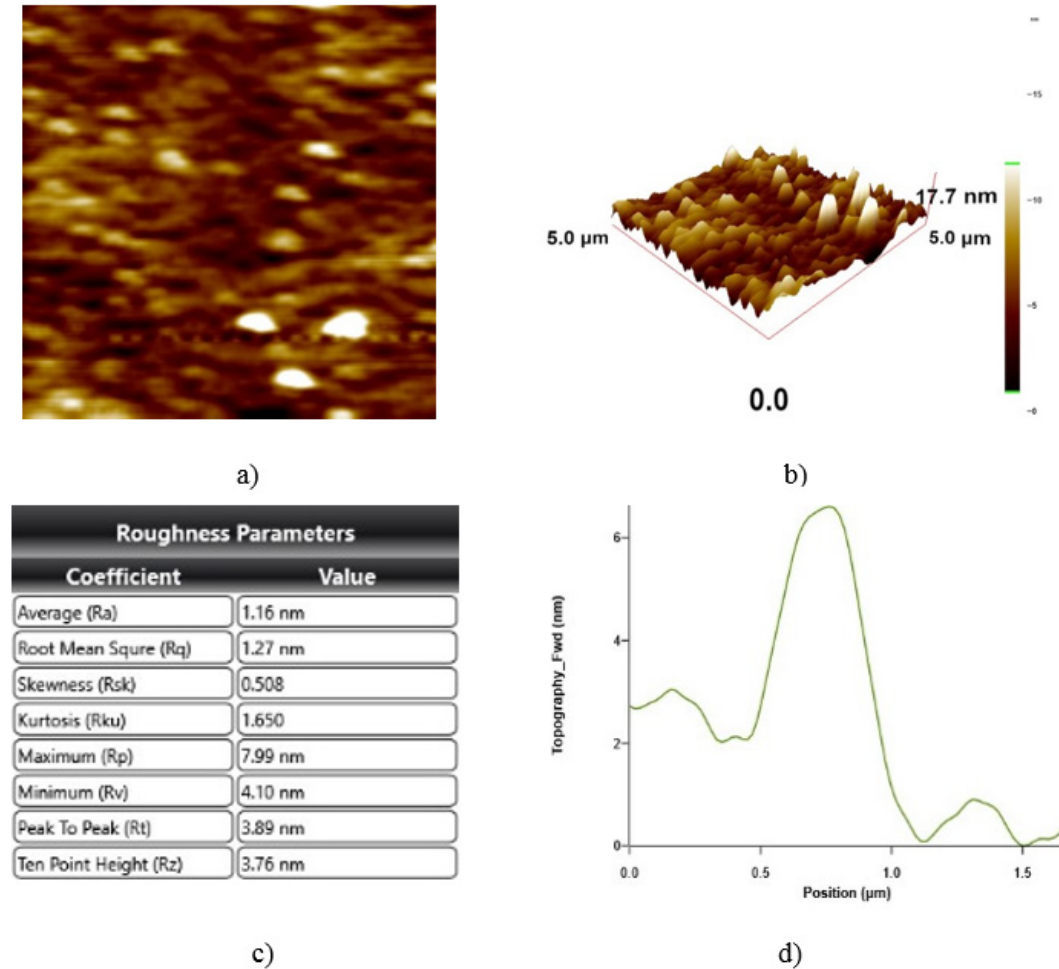


Fig. 4 a) 2-D surface profile of Au nanofilm on silicon substrate, b) 3-D view of same sample, c) average roughness values, and d) roughness of particles in a line

4. Conclusion

After analyzing the results of all the samples in our study, we have developed a basic process for optimizing ezAFM scans. Ultimately, the noise level is the most influential factor in scan quality; however, there are options available to mitigate the impact of high noise on a scan. Sources of this noise include, but are not limited

to, floor noise, electrical noise, and thermal noise. Floor noise is arguably the biggest factor, so setting the vibration isolation platform to the ideal setting is essential. In addition, temperature should remain fairly constant to avoid changes in the sample.

To recap, the relative noise level can be found by performing a scan on the material mica, with $P = 100$, $I = 1$, $D = 100$, and set point = 50. Once the scan is completed, the range of the values in the cross-sectional graph provides an approximation of the noise. Less than 2 Å is ideal. It is possible to observe relative noise by observing the amplitude graph of the scan. If high variations are present in the graph, there is heavy noise, requiring one to decrease the P and D values. If low noise is present, the P and D values should be increased.

Immediately following a scan, it is vital to use the image tools to correct and enhance the image. Plane correction tools allow for the ezAFM program and the viewer to distinguish between different planes on the surface. The line correction tools allow one to remove any stray lines appearing in the scan.

5. References

- Celik U, Celik K, Aslan H, Kehribar I, Dede M, Ozgur Ozer H, Oral A. ezAFM: A low cost atomic force microscope (AFM). American Physical Society March Meeting; Boston, MA; 2012 Feb 27–Mar 2. Vol. 57; p. 1001
- NanoMagnetics Instruments. ezAFM Quick Start Guide. Oxford (UK): NanoMagnetics Instruments; 2014 Feb 3 [accessed 2017 Sep 26]. <https://www.youtube.com/watch?v=YQL1u101kwE>.

List of Symbols, Abbreviations, and Acronyms

2-D	2-dimensional
3-D	3-dimensional
AFM	atomic force microscope
D	derivative gain
ezAFM	Easy Atomic Force Microscope
FT	laser power
I	integral gain
OA	oscillation amplitude
P	proportional gain
PID	proportional, integral, and derivative

1 DEFENSE TECHNICAL
(PDF) INFORMATION CTR
DTIC OCA

2 DIR ARL
(PDF) RDRL DCM
IMAL HRA RECORDS MGMT
RDRL IRB
TECH LIB

1 GOVT PRINTG OFC
(PDF) A MALHOTRA

2 ARL
(PDF) RDRL SEE
S BISOI
RDRL SEE I
N DAS

INTENTIONALLY LEFT BLANK.